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# PATENT COOPERATION TREATY

### **PCT**

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### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 30427	FOR FURTHER ACTION	See Notifi Preliminary	cation of Transmittal of International Examination Report (Form PCT/IPEA/416)		
International application No.	International filing date (day/m	onth/year)	Priority date (day/month/year)		
PCT/US99/11709	27 MAY 1999		28 MAY 1998		
International Patent Classification (IPC) IPC(7): A61L 2/04 and US Cl.: 422/	or national classification and IP6 38, 307, 308	C			
Applicant PEEPLES INDUSTRIES, INC.					
This international prelimina     Examining Authority and is     This REPORT consists of a	transmitted to the applicant a	been prepar	red by this International Preliminary Article 36.		
This report is also accombeen amended and are the (see Rule 70.16 and Sec	This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).				
These annexes consist of a to	otal of sheets.				
3. This report contains indication	ns relating to the following ite	ems:			
I X Basis of the repo	rt				
II Priority					
III Non-establishmer	nt of report with regard to no	velty, invent	ive step or industrial applicability		
IV Lack of unity of	invention				
V X Reasoned statement citations and explain	nt under Article 35(2) with regardations supporting such statem	ard to novelty ent	y, inventive step or industrial applicability;		
VI Certain documents	cited	CC	RRECTED		
VII Certain defects in t	he international application				
VIII X Certain observation	ns on the international application	on .	/ERSION		
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Date of submission of the demand	Date	of completion	n of this report		
24 DECEMBER 1999	02	2 AUGUST 2	000		
Name and mailing address of the IPEA	/US Autho	orized officer	0 111		
Commissioner of Patents and Trader Box PCT		HERESA T.	SNIDER SNIDER		
Washington, D.C. 20231					
Facsimile No. (703) 305-3230	Telep	hone No. (	(703) 308-0661		



### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

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### PCT/US99/11709

1.	Basis of the re	eport			
	Vith regard to the	elements of the international	application:*		
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	the languag	e of a translation furnish e of publication of the ir	this Authority in the following the for the purposes of international application (under the purposes of international for the purpose of the purp	ernational search (und nder Rule 48.3(b)).	
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4.	X The amend	ments have resulted in th	ne cancellation of:		
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* F	beyond the Replacement sheet in this report as	disclosure as filed, as indicates which have been furnished	of) the amendments had not ated in the Supplemental Box to the receiving Office in respect of annexed to this report so	(Rule 70.2(c)).**  conse to an invitation unde	er Article 14 are referred to
	ind 70.17). Anv. rankacamant	sheet containing such ame	endments must be referred to	under item 1 and anne	exed to this report



### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US99/11709

V.	V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement					
1.	statement	_				
	Novelty (N)	Claims Claims	1-18 NONE	_ YES _ NO		
	Inventive Step (IS)	Claims Claims	1-18 NONE	_ YES _ NO		
	Industrial Applicability (IA)	Claims Claims	I-18 NONE	_ YES _ NO		
	cooling of the sterilized slurry by trasnferring	Article 33(2)-( heat to the ur	(4), because the prior art does not teach or fairly suggest t asterilized slurry.	he		
	NONE					
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Form PCT/IPEA/409 (Box V) (July 1998)★

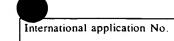




VIII. Certain observations on the international application
The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
Claim 15 is objected to under PCT Rule 66.2(a)(v) as lacking clarity under PCT Article 6 because the claims are indefinite for the following reason(s):
In claim 15, "the first predetermined flow rate" lacks positive antecedent basis since a 'first' flow rate has been previously recited.

Form PCT/IPEA/409 (Box VIII) (July 1998)★

### INTERNATIONAL PRELIMINARY EXAMINATION REPORT



PCT/US99/11709

### Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

Sheet 10

Electrical and the second second

#### I. BASIS OF REPORT:

This report has been drawn on the basis of the description, page(s) 1-8, as originally filed. page(s) NONE, filed with the demand. and additional amendments:

NONE

This report has been drawn on the basis of the claims, page(s) NONE, as originally filed.
page(s) NONE, as amended under Article 19.
page(s) NONE, filed with the demand.
and additional amendments:
Pages 9-12, filed with the letter of 12 June 2000.

This report has been drawn on the basis of the drawings, page(s) 1-3, as originally filed. page(s) NONE, filed with the demand. and additional amendments: NONE

This report has been drawn on the basis of the sequence listing part of the description: page(s) NONE, as originally filed.
pages(s) NONE, filed with the demand.
and additional amendments:
NONE

5. (Some) amendments are considered to go beyond the disclosure as filed: NONE

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### I claim:

- 1. A process for sterilizing a contaminatable slurry, comprising the steps of
  - a. providing a sanitized fluid handling system,
  - b. pumping the slurry through the system at a predetermined pressure,
  - c. heating the slurry to a sterilizing temperature of about 210°F 290°
     for a predetermined time to sterilize the slurry,
  - d. cooling the slurry, and
  - e. transferring the sterilized slurry to a sanitized fluid collection device.
- 2. The process of claim 1, including the step of flushing the fluid handling system with a mist of nitrogen and a sanitizing chemical to sanitize the system.
- 3. The process of claim 1, wherein the sterilizing temperature is about 230°F 270° F, the predetermined time is greater than 5 seconds, and the cooling temperature is below 100° F.
- 4. The process of claim 3, wherein the sterilizing temperature is about 250°F and the predetermined time is about 2 minutes.
- 5. The process of claim 1, wherein the sanitized fluid collection device is a storage tank, and including the steps of
  - f. flushing the fluid handling system with a mist of nitrogen and a sanitizing chemical to sanitize the system, and
  - g. excluding oxygen from the storage tank, and
  - h. transporting the storage tank to a terminus for offloading of the slurry.
- 6. The process of claim 5, including the further steps of
  - i. offloading the slurry, and
  - j. repeating steps a e.

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- 7. The process of claim 1, including the steps of
  - f. flushing the fluid handling system with a mist of nitrogen and a hydrogen peroxide to sanitize the system, and
  - g. excluding oxygen from the tank by covering the sterilized slurry with a blanket of nitrogen.
- 8. The process of claim 1, wherein the fluid collection device is the supply system of a further industrial process.
- 9. The process of claim 1, wherein the slurry is kaolin or calcium carbonate.
- 10. The process of claim 1, including the step of mounting the fluid handling system on a mobile platform for transportability.
- 11. Fluid handling and sterilizing apparatus for sterilizing contaminatable slurries, comprising
  - a sterilizing unit for heating slurry to a sterilizing temperature,
- a heat exchanger for transferring heat from the sterilized slurry to the unsterilized slurry to preheat the unsterilized slurry and cool the sterilized slurry,
  - a piping circuit connecting the pump to the heat exchanger and connecting the heat exchanger to the sterilizing unit for handling the unsterilized slurry, and connecting the sterilizing unit to the heat exchanger and exiting the heat exchanger for handling the sterilized slurry, and
- a pump for intaking unsterilized slurry and pumping slurry through the piping circuit at a predetermined pressure.
  - 12. The fluid handling and sterilizing apparatus of claim 11, wherein the sterilizing unit includes
    - a heating unit,

- a second heat exchanger for transferring heat from the heating unit to the
  unsterilized slurry to raise the temperature of the unsterilized slurry to a
  sterilizing temperature,
  - a manifold for containing the heated slurry at the sterilizing temperature, and
- a third heat exchanger for transferring heat from the sterilized slurry to the heating unit to cool the sterilized slurry.
  - 13. The fluid handling and sterilizing apparatus of claim 12, wherein the manifold is sized to maintain the slurry at the sterilizing temperature for a predetermined time sufficient to fully sterilize the slurry.
  - 14. The fluid handling and sterilizing apparatus of claim 12, wherein the heating unit comprises
  - a hot water boiler for heating water to a second predetermined temperature,
  - a second piping circuit interconnecting the hot water boiler, the second heat exchanger, and the third heat exchanger, and
    - a second pump for pumping water through the second piping circuit at a second predetermined flow rate.
    - 15. The fluid handling and sterilizing apparatus of claim 14, wherein the first predetermined flow rate is about 17 psig, and the second predetermined flow rate is about 30 psig.
  - 5 16. The fluid handling and sterilizing apparatus of claim 11, wherein the contaminatable slurry is kaolin or calcium carbonate.
    - 17. The fluid handling and sterilizing apparatus of claim 11, where the pump is a peristaltic pump.

18. The fluid handling and sterilizing apparatus of claim 11, including a mobile platform mounting the apparatus to enable transportation to a variety of locations.

### PCT

## WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



### INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(74) Agent: GREENLEE, David, A.; P.O. Box 340557, Columbus, OH 43234-0557 (US).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

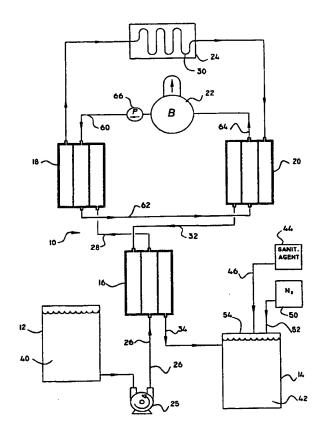
#### **Published**

With international search report.

(54) Title: SLURRY TREATMENT AND SHIPPING METHOD

### (57) Abstract

Aerobic and anaerobic contamination is removed from a slurry by a sterilizing process and apparatus (10). A slurry piping circuit (26, 28, 30, 32, 34) interconnects a supply tank (12), three serial heat exchangers (16, 18, 20) and a storage tank (14). The circuit and storage tank are sanitized by flushing with mist of hydrogen peroxide (44) and nitrogen One heat exchanger (16) transfers heat from the sterilized slurry to the unsterilized slurry. A closed hot water piping circuit (60, 62, 64) interconnects a hot water boiler (22) and the other two heat exchangers (18, 20). The slurry is sterilized in an insulated manifold (24) interconnecting the other two heat exchangers (18, 20). Slurry is pumped through the piping and heated by hot water in the manifold to kill the bacteria, and is covered by a layer of nitrogen (54) in the storage tank (14). The storage tank may be transported to a distant terminus where it is unloaded and again sterilized, if necessary, or is used immediately in an industrial process.



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### INTERNATIONAL SEARCH REPORT

International application No. PCT/US99/11709

A. CLASSIFICATION OF SUBJECT MATTER  IPC(6) :A61L 2/04  US CL :422/38, 307, 308								
According to	According to International Patent Classification (IPC) or to both national classification and IPC							
	DS SEARCHED	1 1 25						
Minimum do	ocumentation searched (classification system followed	by classification symbols)						
	122/38, 307, 308							
Documentati NONE	Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched NONE							
Electronic d	ata base consulted during the international search (nar	me of data base and, where practicable	, search terms used)					
APS, CAI								
C. DOC	UMENTS CONSIDERED TO BE RELEVANT							
Category*	Citation of document, with indication, where app	propriate, of the relevant passages	Relevant to claim No.					
Y	US 3,871,824 A (RECHTSTEINER	ET AL) 18 March 1975	2, 5-7					
	(18.03.75), see col.4, lines 26-35; col.	7, lines 7-15.						
A		1	15					
Y	US 5,225,221 A (CAMDEN ET AL) 06 July 1993 (06.07.93), see col.7, line 59 and col.9, lines 58-60.							
x	US 5,498,396 A (AIKUS ET AL) 12 entire document.	March 1996 (12.03.96), see	1,3,4,8,10					
X,P	US 5,759,491 A (BUNIN) 02 June document.	1998 (02.06.98), see entire	11-13, 16-18					
Y,P	US 5,888,453 A (LUKER) 30 March document.	1999 (30.03.99), see entire	14					
Furti	her documents are listed in the continuation of Box C	. See patent family annex.						
1	pecial categories of cited documents:	*T* later document published after the int date and not in conflict with the app the principle or theory underlying th	lication but cited to understand					
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	e actual completion of the international search	Date of mailing of the international se	earch report					
16 AUG	UST 1999	10 SEP 1999						
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(75) Inventor/Applicant (for US only): ADLER, Paul, E. [US/US]; 103 Georgetown Court, Macon, GA 31210 (US).

(74) Agent: GREENLEE, David, A.; P.O. Box 340557, Columbus, OH 43234-0557 (US).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

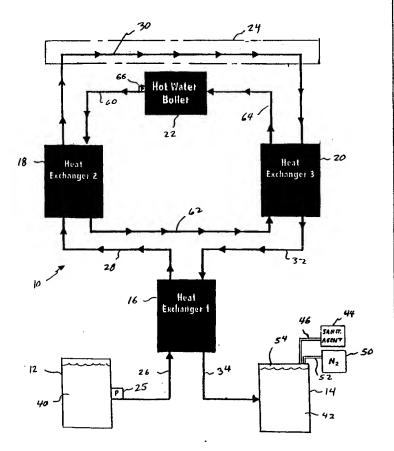
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### SLURRY TREATMENT AND SHIPPING METHOD

This invention relates generally to treating contaminatable slurries and, more particularly, to a process and apparatus for sterilizing and a method of shipping these slurries.

TECHNICAL FIELD

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### BACKGROUND OF THE INVENTION

Many processes exist for treating mined kaolin during the beneficiation process to improve the physical and chemical aspects of the resulting slurry, or slip, to improve its commercial value. Many of these remove contaminants of various types to improve the whiteness and purity of the kaolin and additionally employ the use of oxidation, leaching and magnetic separation. Such processes are detailed in United States Patent No. 5,397,754.

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Slurries of beneficiated kaolin, calcium carbonate, titanium dioxide, talc, latex, other carbonates and mixtures thereof are used extensively in the paper, paint, rubber and plastics industries as coatings, fillers, extenders and pigments. These slurries are contaminatable (i.e. subject to contamination) by aerobic and anaerobic bacteria. Such contamination affects the color, odor and viscosity of the slurries, which negatively affect their commercial value.

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Many treatments to neutralize the effects of this bacteria on kaolin slurries have been tried. The most conventional treatment is mixing a bactericide into the slurry to kill the bacteria. An example of this treatment is found in United States Patent No. 5,496,398. Unfortunately, this treatment has a very limited period of effectiveness until contamination again occurs, because the

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bacteria are not completely destroyed and exposure to air produces recontamination by the aerobic bacteria that thrive on the available oxygen. Thus, the slurry must be re-treated if it is not quickly used. This repeated use of such chemicals increases the cost of producing and maintaining these slurries at acceptable levels of bacteriological contamination while assuring that the performance properties of the slurries are not degraded.

The limited life of such a treatment process presents a problem when contaminatable slurries are stored for long periods of time, a problem that is exacerbated by the conventional practice of storing slurries in open top tanks. Often, the treated slurries are shipped for long distances by truck, rail or ship, which produce re-contamination for the same reasons.

There is a need for a method of treating and shipping kaolin and calcium carbonate and other contaminatable slurries which eliminates bacterial contamination during storage and shipping and eliminates the necessity for repeated treatment by bactericides.

### **SUMMARY OF THE INVENTION**

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It is therefore an object of this invention to provide for a method of treating and shipping kaolin and calcium carbonate and other contaminatable slurries which eliminates bacterial contamination during storage and shipping and eliminates the necessity for repeated treatment by bactericides.

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According to a preferred embodiment of this invention, apparatus is provided for sterilizing slurries subject to aerobic and anaerobic contamination. A slurry piping circuit interconnects a supply tank, three serial heat exchangers, and a storage tank. The circuit and storage tank are sanitized by flushing with hydrogen peroxide. One heat exchanger transfers heat from the sterilized slurry to the unsterilized slurry. A closed hot water piping circuit interconnects a hot

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water boiler and the other two heat exchangers. The piping interconnecting the other two heat exchangers is an insulated manifold where the slurry is sterilized. Slurry is pumped through the piping and heated by hot water in the manifold to kill the bacteria, and is stored and covered by a layer of nitrogen. It may be transported to a distant terminus where it is unloaded and again sterilized, if necessary, or be stored locally, or be used immediately in an industrial process.

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In one aspect, this invention features a process for sterilizing contaminatable slurries, which comprises the steps of providing a sanitized fluid handling system, pumping the slurry through the system at a predetermined flow rate, heating the slurry to a sterilizing temperature of about 210°F - 290° for a predetermined time to sterilize the slurry, cooling the slurry to a temperature below 100°F, and transferring the sterilized slurry to a sanitized, oxygen-free fluid collection device, which may be a stationary or transportable storage tank or the supply system for a further industrial process.

In another aspect of this invention, the process includes the further steps of excluding oxygen from the tank by covering the sterilized slurry with a blanket of nitrogen, and transporting the storage tank to a terminus for offloading of the slurry and re-sterilizing if necessary.

In yet another aspect, this invention features fluid handling and sterilizing apparatus for sterilizing contaminatable slurries, which includes a supply tank for holding unsterilized slurry, and a sterilizing unit for heating slurry to a sterilizing temperature. A heat exchanger transfers heat from the sterilized slurry to the unsterilized slurry, thus preheating the unsterilized slurry and cooling the sterilized slurry. A slurry piping circuit connects the supply tank to the heat exchanger and connects the heat exchanger to the sterilizing unit for handling the unsterilized slurry, and connects the sterilizing unit to the heat exchanger and exits the heat exchanger for handling the sterilized slurry. A pump pumps slurry

through the piping circuit at a predetermined pressure in order to maintain all slurry in a liquid state, regardless of temperature.

In a further aspect of this invention, the sterilizing unit includes a heating unit, a second heat exchanger for transferring heat from the heating unit to the unsterilized slurry to raise the temperature of the unsterilized slurry to a sterilizing temperature, a manifold for maintaining the heated slurry at the sterilizing temperature, and a third heat exchanger for transferring heat from the sterilized slurry to the heating unit to cool the sterilized slurry.

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These and further objects and features of this invention will become readily apparent from a review of the following detailed description of a preferred embodiment, as illustrated in the accompanying drawings, in which:

### **BRIEF DESCRIPTION OF THE DRAWINGS**

Fig. 1 is a schematic representation of the apparatus and process according to this invention;

Fig. 2 is a simplified version of the schematic of Fig. 1, depicting system temperatures measured during successful operation of a sterilizing system according to this invention; and

Figs. 3 and 4 are plan and side views of the system of Fig. 1, shown mounted on a semi-trailer.

### DESCRIPTION OF THE PREFERRED EMBODIMENT

Figs. 1 and 2 of the drawings schematically depict the major components of a system 10 for continuously sterilizing contaminatable slurry,

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according to a preferred embodiment of this invention. These comprise a supply tank 12, a storage tank 14, serially arranged heat exchangers 16, 18 and 20, a hot water boiler 22, and a manifold 24.

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A pump 25 at supply tank 12 pumps slurry through a piping system, which includes a supply pipe 26 from pump 25 to heat exchanger 16, a connecting pipe 28, that connects heat exchangers 16 and 18, slurry manifold piping 30, that connects heat exchangers 18 and 20, a connecting pipe 32, that connects heat exchangers 20 and 22, and an outlet pipe 34, that exits heat exchanger 16 and enters storage tank 14. The direction of slurry flow is indicated by arrows in Figs. 1 and 2. Pipe 34 can, alternatively, connect directly into the intake of a further industrial process, such as paper making. Tanks 12 and 14 may be stationary, or mobile, such as mounted on a rail car, semi-trailer, or ship. Indeed, the slurry sterilizing system itself may be stationary or may be mounted on a similar mobile platform for transportation to a variety of locations for on-site processing (e.g. at an industrial plant, at dockside, or in a railroad yard).

At the initiation of the process of this invention, supply tank 12 will be filled with contaminated, or unsterilized, slurry 40, while storage tank 14 will normally be empty. This process is a continuous process, so that eventually tank 14 will fill with sterilized slurry 42. Prior to initiation of this process, at least tank 14, and pipes 34, 32, and 30, and preferably pipes 28 and 26 will be sanitized by backflushing with a sanitizing agent, such as hydrogen peroxide, supplied from a supply 44 via a delivery nozzle 46. Oxygen is purged from the system by the simultaneous introduction of nitrogen from a supply 50 via a nozzle 52.

The heating unit for the sterilizing apparatus 10 includes hot water boiler 22, and a second or hot water piping system, that includes an outlet pipe 60 from boiler 22 to heat exchanger 18, a connecting pipe 62 connecting heat exchangers 18 and 20, and an inlet pipe 64 connecting heat exchanger 20 to boiler 22. A

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pump 66 pumps water through the closed hot water system at a predetermined pressure in the direction indicated by arrows in the drawings. The pressure of the hot water system must be sufficient to preclude formation of steam. The heating unit and manifold 24 comprise the sterilizing unit.

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A prototype/pilot sterilization system, illustrated in Figs. 3 and 4, was constructed on a semi-trailer 70 to test and prove out the process according to this invention. This system utilized a Parker manifold comprising 60 ft. of 4" stainless steel slurry pipe and an array of 2" stainless steel hot water pipe - all enclosed in a stainless steel box containing 3" rock wool insulation. The heating system utilized a 300,000 BTU/hr. propane-fired boiler and a pumping system that pumped hot water at 30 gpm. A peristaltic slurry pump pumped slurry through the system at 17 gpm. Three substantially identical APV plate heat exchangers were interconnected by insulated piping runs of 10 ft. A 35 kW Cummins generator set powered the installation.

A target sterilizing temperature was set at about 250° F and a holding time of about 2 minutes in the sterilizing manifold. The sterilizing temperature was actually measured at 255° F and the time was about 2 minutes. A preferred temperature range is about 230° F - 270° F, with the time of treatment being longer than 5 seconds for a flash treatment. Treatment time is dependent on the volume of the piping used and treatment temperature. The temperature of the cooled sterilized slurry was 90° F, and a temperature below 100° F is preferred to facilitate handling.

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The system was fogged with a mist composed of a mixture of nitrogen and hydrogen peroxide emitted from tanks through a dual nozzle to sanitize the system. After the system was fired up and brought to temperature, 700 gallons of production beneficiated calcium carbonate slurry, at 75% dry solids and a density of 16 pounds/gallon volume, were pumped through the sanitized system. Temperatures in all segments of the system were monitored, as was water flow.

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Temperatures measured during this run are depicted in Fig. 2. Samples were taken of sterilized slurry to confirm that sterilization was complete. The test was successful, thus proving the process. It is now possible to sterilize contaminatable slurries without using bactericides or other chemicals.

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This process proved to be quite economical, as the heating system recovered 90% of the boiler heat. Thus, only 10% boiler makeup is necessary. This economy results from the use of heat exchangers to transfer heat. As Fig. 2 shows, the incoming 80° F contaminated slurry was pre-heated to 165° F in heat exchanger 16 by the 180° F sterilized slurry, which cooled to an exit temperature of 90° F. The preheated contaminated slurry was elevated to a sterilizing temperature in heat exchanger 18 by 265° F hot water from boiler 22 to a temperature of 255° F, as measured in manifold 24. Hot water, which exited manifold 24 at 173° F, was reheated to 247° F in heat exchanger 20 by sterilized slurry, which then exited at 180° F. Thus the boiler was supplied with 247° F intake water, which it needed to elevate only 18° F.

Preferably, a mist or fog of nitrogen and hydrogen peroxide is used to sanitize the system. Sanitizing chemicals other than hydrogen peroxide may also be used to kill any bacteria in the system. As slurry from supply 40 transits slurry piping 26, 28, 30, 32 and 34 into storage tank 14, this fog is forced into storage tank 14, where the condensed hydrogen peroxide will be drained and the nitrogen will blanket slurry 42 to exclude contact with oxygen

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The nitrogen blanket is maintained during any subsequent shipping of tank 14 to a distant terminus by truck, rail or ship. Depending on the degree of sterilization maintained (a function of time and equipment), the transported slurry may again undergo the same sterilization process by another or similar installation of sterilizing equipment 10.

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It is understood that use of the term "contaminatable slurries" herein encompasses any slurry of substances, such as kaolin, calcium carbonate, titanium dioxide, talc, latex, other carbonates, and combinations thereof, which are subject to contamination by aerobic and anaerobic bacteria, and which are used as coatings, fillers, extenders and pigments in the paper, paint, rubber and plastics industries.

While only a preferred embodiment has been illustrated and described, obvious modifications are contemplated within the scope of this invention, as defined by the appended claims. For example, other types of heating systems could be used to sterilize the slurry, although systems using a liquid are preferred because of their efficiency.

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### I claim:

- 1. A process for sterilizing a contaminatable slurry, comprising the steps of
  - a. providing a sanitized fluid handling system,
  - b. pumping the slurry through the system at a predetermined pressure,
  - c. heating the slurry to a sterilizing temperature of about 210°F 290°
     for a predetermined time to sterilize the slurry,
  - d. cooling the slurry, and
  - e. transferring the sterilized slurry to a sanitized fluid collection device.
- 2. The process of claim 1, including the step of flushing the fluid handling system with a mist of nitrogen and a sanitizing chemical to sanitize the system.
- 3. The process of claim 1, wherein the sterilizing temperature is about 230°F 270° F, the predetermined time is greater than 5 seconds, and the cooling temperature is below 100° F.
- 4. The process of claim 3, wherein the sterilizing temperature is about 250°F and the predetermined time is about 2 minutes.
- 5. The process of claim 1, wherein the sanitized fluid collection device is a storage tank, and including the steps of
  - f. flushing the fluid handling system with a mist of nitrogen and a sanitizing chemical to sanitize the system, and
  - g. excluding oxygen from the storage tank, and
  - h. transporting the storage tank to a terminus for offloading of the slurry.
- 6. The process of claim 5, including the further steps of
  - i. offloading the slurry, and
  - j. repeating steps a e.

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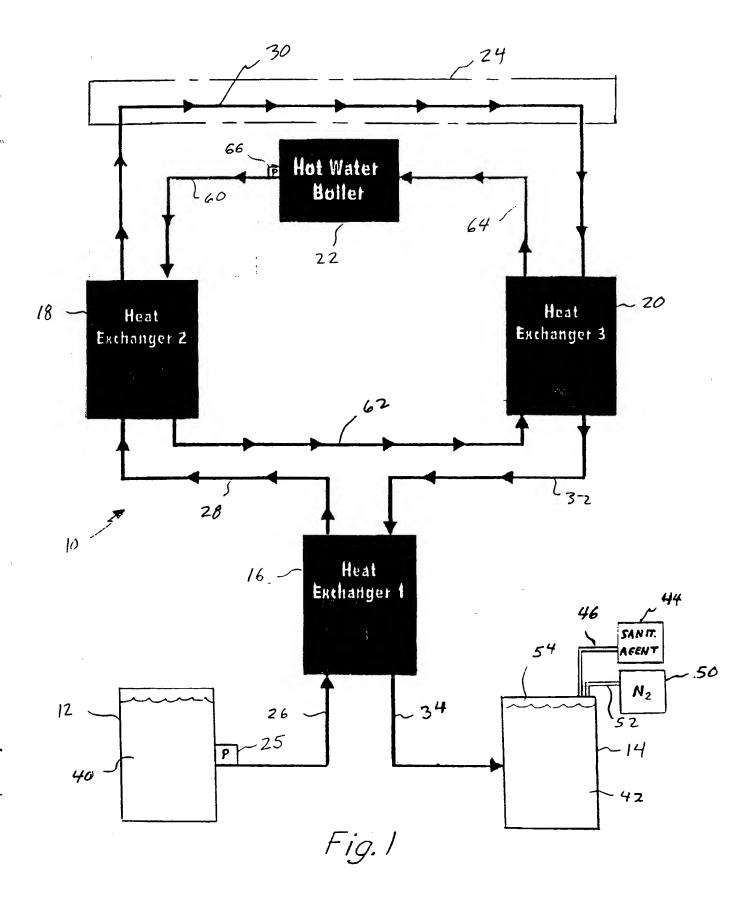
- 7. The process of claim 1, including the steps of
  - f. flushing the fluid handling system with a mist of nitrogen and a hydrogen peroxide to sanitize the system, and
  - g. excluding oxygen from the tank by covering the sterilized slurry with a blanket of nitrogen.
- 8. The process of claim 1, wherein the fluid collection device is the supply system of a further industrial process.
- 9. The process of claim 1, wherein the slurry is kaolin or calcium carbonate.
- 10. The process of claim 1, including the step of mounting the fluid handling system on a mobile platform for transportability.
- 11. Fluid handling and sterilizing apparatus for sterilizing contaminatable slurries, comprising
  - a sterilizing unit for heating slurry to a sterilizing temperature,
- a heat exchanger for transferring heat from the sterilized slurry to the unsterilized slurry to preheat the unsterilized slurry and cool the sterilized slurry,
- a piping circuit connecting the pump to the heat exchanger and connecting the heat exchanger to the sterilizing unit for handling the unsterilized slurry, and connecting the sterilizing unit to the heat exchanger and exiting the heat exchanger for handling the sterilized slurry, and
- a pump for intaking unsterilized slurry and pumping slurry through the piping circuit at a predetermined pressure.
  - 12. The fluid handling and sterilizing apparatus of claim 11, wherein the sterilizing unit includes
    - a heating unit,

a second heat exchanger for transferring heat from the heating unit to the unsterilized slurry to raise the temperature of the unsterilized slurry to a sterilizing temperature,

a manifold for containing the heated slurry at the sterilizing temperature, and

- a third heat exchanger for transferring heat from the sterilized slurry to the heating unit to cool the sterilized slurry.
  - 13. The fluid handling and sterilizing apparatus of claim 12, wherein the manifold is sized to maintain the slurry at the sterilizing temperature for a predetermined time sufficient to fully sterilize the slurry.
  - 14. The fluid handling and sterilizing apparatus of claim 12, wherein the heating unit comprises
  - a hot water boiler for heating water to a second predetermined temperature,
  - a second piping circuit interconnecting the hot water boiler, the second heat exchanger, and the third heat exchanger, and
    - a second pump for pumping water through the second piping circuit at a second predetermined flow rate.
    - 15. The fluid handling and sterilizing apparatus of claim 14, wherein the first predetermined flow rate is about 17 psig, and the second predetermined flow rate is about 30 psig.
  - 5 16. The fluid handling and sterilizing apparatus of claim 11, wherein the contaminatable slurry is kaolin or calcium carbonate.
    - 17. The fluid handling and sterilizing apparatus of claim 11, where the pump is a peristaltic pump.

18. The fluid handling and sterilizing apparatus of claim 11, including a
5 mobile platform mounting the apparatus to enable transportation to a variety of locations.



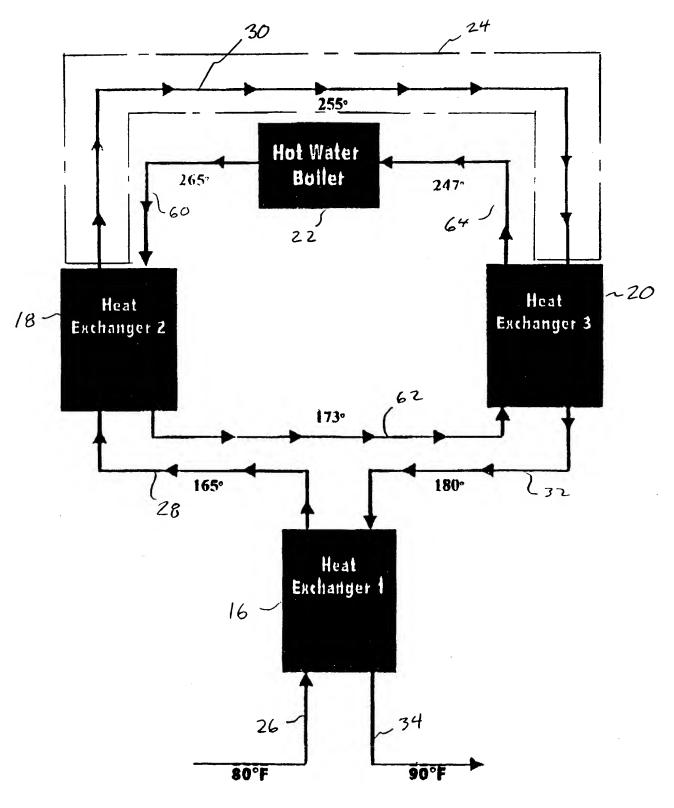
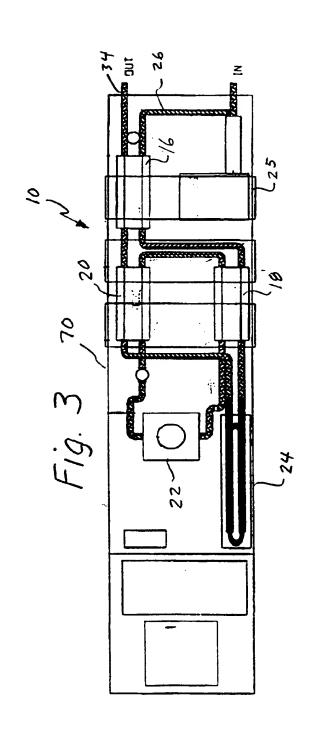
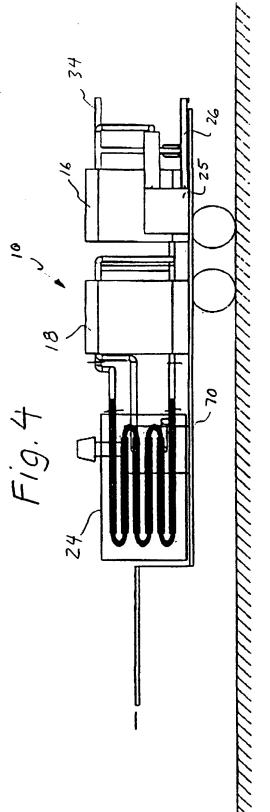


Fig. 2







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### INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 30427		Transmittal of International Search Report 0) as well as, where applicable, item 5 below.
International application No.	International filing date (day/month/year)	(Earliest) Priority Date (day/month/year)
PCT/US99/11709	27 MAY 1999	28 MAY 1998
Applicant PEEPLES INDUSTRIES, INC.		
	ts of a total of $\frac{1}{2}$ sheets.	athority and is transmitted to the applicant
	copy of each prior art document cited in this	report.
1. Certain claims were found	unsearchable (See Box I).	
2. Unity of invention is lacking	ng (See Box II).	
	n contains disclosure of a nucleotide and/oried out on the basis of the sequence listing	or amino acid sequence listing and the
	filed with the international application.	
	furnished by the applicant separately from the	international application,
		nent to the effect that i. did not include matter the international application as filed.
	transcribed by this Authority.	
4. With regard to the title, X	the text is approved as submitted by the appli	cant.
. 🗆	the text has been established by this Authority	to read as follows:
5. With regard to the abstract,		
X	the text is approved as submitted by the appli	cant.
. 🗆	the text has been established, according to Rule Box III. The applicant may, within one month ( search report, submit comments to this Autho	rom the date of mailing of this international
6. The figure of the drawings to be	published with the abstract is:	
Figure No. 1 X	as suggested by the applicant.	N co r
	because the applicant failed to suggest a figur	None of the figures.
	because this figure better characterizes the in-	vention.

International application No. PCT/US99/11709

A. CLASSIFICATION OF SUBJECT MATTER IPC(6) :A61L 2/04								
	US CL :422/38, 307, 308  According to International Patent Classification (IPC) or to both national classification and IPC							
h	DS SEARCHED							
Minimum d	locumentation searched (classification system follower	ed by classification symbols)						
U.S. :	422/38, 307, 308							
Documental NONE	Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched NONE							
Electronic o	Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)							
APS, CA search ter	PLUS rms: slurry, disinfect?, steril?							
C. DOC	CUMENTS CONSIDERED TO BE RELEVANT							
Category*	Citation of document, with indication, where a	ppropriate, of the relevant passages	Relevant to claim No.					
Y	US 3,871,824 A (RECHTSTEINER		2, 5-7					
	(18.03.75), see col.4, lines 26-35; col	.7, lines 7-15.	15					
A			15					
Y	US 5,225,221 A (CAMDEN ET AL) col.7, line 59 and col.9, lines 58-60.	06 July 1993 (06.07.93), see	•)					
x	US 5,498,396 A (AIKUS ET AL) 12 entire document.	March 1996 (12.03.96), see	1,3,4,8,10					
X,P	US 5,759,491 A (BUNIN) 02 June document.	1998 (02.06.98), see entire	11-13, 16-18					
Y,P	US 5,888,453 A (LUKER) 30 March document.	1999 (30.03.99), see entire	14					
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Furth	her documents are listed in the continuation of Box C	C. See patent family annex.						
*A* do	pecial categories of cited documents:  Decument defining the general state of the art which is not considered be of particular relevance.	"T" later document published after the inte date and not in conflict with the appl the principle or theory underlying the	ication but cited to understand					
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